LA-UR-21-24786
Approved for public release; distribution is unlimited.

Title: Protocols for Uranium Carbon Analysis - Testing Protocol Summary

Author(s): Tubesing, Philip K.

Intended for: Report

Issued: 2021-05-18

Los Alamos
NATIONAL LABORATORY

Protocols for Uranium Carbon Analysis – Testing Protocol Summary

Philip K. Tubesing
Sigma-DO

April 2021

LA-UR-21-XXXXX

Carbon Analysis in Uranium Protocol History

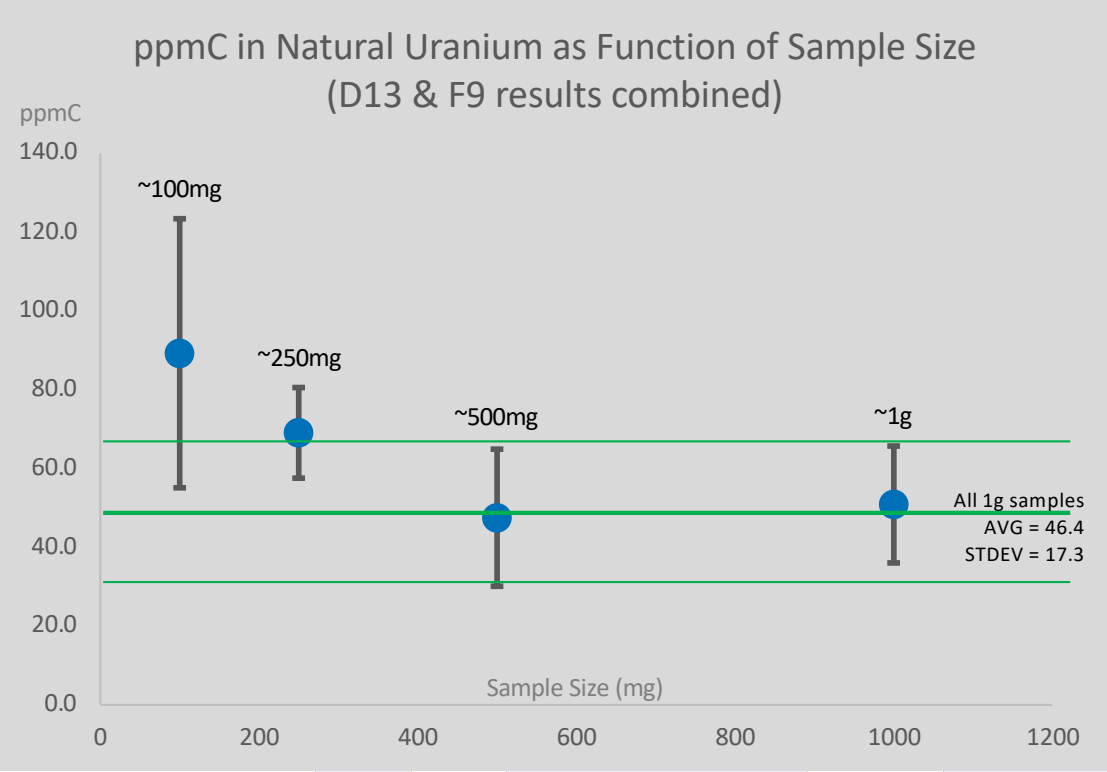
- In 1998, Sigma (then MST-6) purchased and installed a new carbon-sulfur analyzer in G105 as part of a substantial refresh of our analytic capabilities.
  - Horiba EMIA-8200W Carbon/Sulfur Analyzer

- Substantial effort was undertaken to establish analysis protocols for uranium and other materials. This summary describes the following protocols we put in place as a result of these studies.
  - Sample Size (for monolithic samples—chip and powder sample may need more current study)
  - Flux/Accelerant Recipe
  - Sample Cleaning Process

This summary is extracted from the protocol development description detailed in LA-UR-21-23989

Uranium Sample Size Protocol for Carbon Analysis

Based upon the results of >100 samples from a natural uranium carbon analysis round robin, we established ~500mg as the lower threshold for sample size.

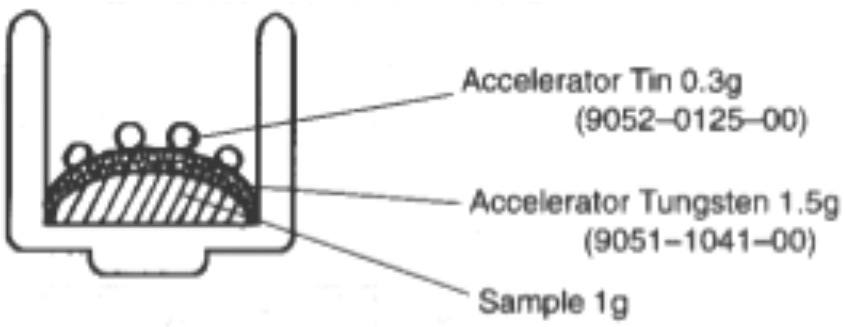


These results are for monolithic samples.
Some work was done on uranium chips & turnings, but not enough to establish sample size thresholds.

Los Alamos NATIONAL LABORATORY

Derived from LA-UR-21-23989

Fluxing Recipe for Uranium Carbon Analysis



Flux (accelerant) recipe Sigma used for nearly all carbon analysis samples (of any sample material) [1]

Sample + 1.5g W + 0.3g Sn

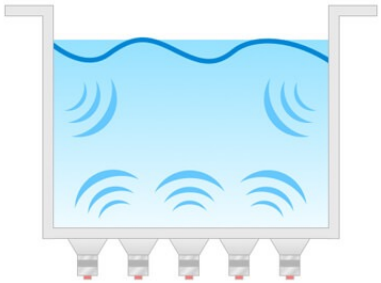
2.0g W + 0.5g Sn was tried. Our recollection is that this caused unacceptable crucible boil over, and splattering of the furnace tube interior.

0.5g W + 0.1g Sn "Light Flux" was also tried for samples ~0.25g and less. This appeared to improve the accuracy of the average values, but significantly increased the spread of the data.

[1] Image taken from Horiba EMIA-8200W Instruction Manual, Second Edition, Horiba LTD, December 1997, Code I042935100, Page 51

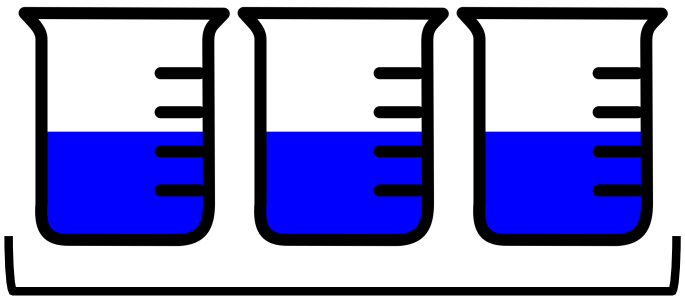
Los Alamos NATIONAL LABORATORY

Uranium Sample Preparation Protocol for Carbon Analysis



Soapy Water
Ultrasonic bath
60 sec

10% Dilute
Nitric Acid
90 sec [1]



3-Step De-Ionized
Water Rinse Cascade
20 sec each

Acetone
Rinse
5-10 sec [2]

Dry with
"Warm Wind" [3]

[1] A 5:1 Concentrated Nitric Acid cleaning for 15-20 seconds was also used on occasion

[2] If fully dried/evaporated from the sample surface, acetone was shown not to affect the carbon results

[3] This term was taken from one of the Horiba EMIA manuals. We utilized a standard commercial electric heat gun to dry the samples.

Los Alamos
NATIONAL LABORATORY

Derived from LA-UR-21-23989